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SUBJECT: Test of a Natural-Circulating High-Pressure Recombiner for Hydrogen and Oxygen
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ABSTRACT

A natural-circulation loop to catalytically recombine hydrogen and oxygen at high pressure was built and operated without incident; there were no explosions and no failures of the Inconel system. Stoichiometric $2H_2 + O_2$ electrolytically generated from 15% KOH at high pressure, was recombined on platinized wire mesh at a rate of 0.60 - 0.75 scfm at 350°C and 1500 psi total pressure for a period of 2500 hours. The concentration of stoichiometric gas ranged up to 8 mol %, the diluent being steam and excess O_2 .

Metallurgical examination of zirconium and titanium alloys and a modified 430 stainless steel exposed in the system indicated none would be a suitable material of construction for all parts of the recombinder system, although Zircaloy-2 would be adequate for fabrication of the boiler, and the modified 430 stainless steel for the recombinder section.

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INTRODUCTION

Radiolytic hydrogen and oxygen produced in the aqueous homogeneous reactor can be recombined in the high pressure gas phase on a solid catalyst, thereby greatly minimizing the amount of gas and liquid which must be let down and subsequently pumped back to high pressure.

Recent work on the forced-circulation high pressure recombiner established mass transfer rates, demonstrated a suitable catalyst-catalyst support, and established that explosions can be prevented.¹

The natural circulation recombiner was built to test the feasibility of operation on a natural convection cycle, which eliminates the complication of a pump-ejector combination or high pressure blower for moving the gas. Also, Inconel was used as a primary material of construction.

Primary objectives of the natural circulation loop were:

- (1) Determine the general characteristics of such a system, e.g., steady state knallgas concentration, necessity for excess oxygen, relationship among saturation temperature, bed outlet temperature, cooler outlet temperature, gas flow rate, etc.
- (2) Observe the control characteristics of the system.
- (3) Demonstrate continuous, reliable operation.

Secondary objectives were:

- (1) Demonstrate that Inconel is a satisfactory material of construction for the environment: KOH, steam, H₂, and O₂ at 300 - 350°C.
- (2) Continue the exposure of a modified 430 stainless steel (Croloy 16-1) and alloys of zirconium and titanium.
- (3) Demonstrate the long-term reliability of the platinized mesh catalyst.

GENERAL DESCRIPTION - OPERATION AND CONTROL

Operation

Figure 1 shows the entire high pressure system, consisting of a natural convection loop of 2 in. Inconel pipe welded to a boiler fabricated from 6 in. Inconel pipe. Immediately below the boiler are twin electrolytic cells, fabricated of nickel-plated carbon steel pipe, which generate up to 1 scfm of stoichiometric 2H₂ + O₂ (knallgas) from 8-15% KOH solution. Knallgas rises from the cells, through the boiler liquid, into the continuous gas phase where it mixes with the recirculating stream of superheated steam, unreacted knallgas, and excess oxygen before entering the recombiner bed. In passing through the 9 in. long catalyst roll of platinized stainless wire mesh, part of the knallgas recombines to produce an effluent mixture at an elevated temperature. Air coolant

Fig. 1. High Pressure Recombiner, Including Boiler and Electrolytic Cells.

on the second leg of the cycle increases the gas density within the leg to provide a driving force for convection.

Control

Figure 2 depicts the control instruments used, and their functions. Three Minneapolis-Honeywell controllers of 400°C range controlled electrical heat input to the boiler and the superheater, and regulated the cooling air. Safety interlocks provided automatic loop shutdown when temperature, pressure, amperage, or liquid level moved outside specified limits. See Appendix A.

Start-up

With the loop filled as indicated in Figure 1, the system was evacuated, the boiler was brought to about 310°C, and oxygen was injected to the desired partial pressure (generally about 20 psia). The cooler was set to control the cooler effluent at 2 to 5°C superheat, while the superheater was cut in at a nominal rate of 1.4 kw. When the system temperatures stabilized, knallgas generation was begun at about 250 amperes and increased rapidly to 1750 amp in steps of 250 to 500 amp. Pressure increase, after each increase in amperage, was allowed to "peak" before each succeeding increase. When catalyst bed outlet temperature reached 350°C, control of electrical input to the superheater was changed to automatic to maintain the outlet temperature at this value. At 1750-1800 amperes and above, no electrical superheat was required to maintain the catalyst bed outlet temperature at 350°C or above.

RESULTS

Operation of the natural circulation recombiner for over 2500 hrs demonstrated the general reliability of the system.

Early operation of the loop was limited by boiler heating capacity and by air coolant capacity. Electrical failure early in the run of one electrolytic cell reduced knallgas generation capacity to 2000 amperes. Appendix B is a condensed log of operations; Table 1 summarizes operating conditions for most of the 2500 hrs.

Table 1. Operating Conditions

$P_{\text{total}} = 1547 \text{ psia}$	
$P_{\text{excess O}_2} = 20 \text{ psia}$	
$T_{\text{saturation}} = 308.5^\circ\text{C}$	
$I_{\text{cells}} = 1810 \text{ amperes}$	2/3 scfm knallgas
<u>Gas Entering Bed</u>	<u>Gas Leaving Bed</u>
Composition* (mol %) 5.4% H ₂ 3.9% O ₂ , 90.7% H ₂ O	4.3% H ₂ , 3.4% O ₂ , 92.3% H ₂ O
Flow rate* 50 gm moles/min	49.7 gm moles/min = 0.75 ft ³ /min
Velocity*	0.61 ft/sec
Temperature 315°C	350°C

*Values obtained by trial-and-error energy balance. See Appendix C.

Fig. 2. Schematic of Temperature Control Arrangement.

Knallgas Concentration

As indicated in Table 1, knallgas concentration in the stream entering the catalyst bed was about 8 mol. % under long term steady-state conditions. A gas sample taken under somewhat the same conditions indicated a concentration of 7.2%.

It was not possible to calculate accurately the knallgas concentration during unsteady-state conditions because the saturated steam pressure was strongly affected by the effective KOH concentration in the boiler section. KOH concentration, as determined by chemical analysis, ranged from 18 to 4 wt % - corresponding to a variation in saturated steam pressure of approximately 200 psia.

Concentration of stoichiometric gas was inferred to be little affected by changes in gas generation rate within the capacity of one electrolytic cell, with constant electrical input to the superheater section. See Table 2 and Figure 3. As expected, the resulting increase in temperature difference between hot and cold legs tended to increase the gas flow rate and thereby minimize the increase in knallgas concentration.

Table 2. Bed Outlet Temperature Related to Electrolysis Current

($T_{\text{boiler}} = 315^{\circ}\text{C}$, Pressure excess $\text{O}_2 = 130$ psia. Time meter reference: 269.3 - 272.2 hrs)

Amperage, Electrolytic Cells	Temperature Bed Outlet °C	Temperature Cooler Outlet °C	Pressure Total psia
0	327	317	1590
370	333	316	1610
700	337	315	1610
1400	348	315	1635

Superheat Effect on Control Characteristics

Control of loop pressure, flow rate, etc., was most strongly influenced by the cooler outlet temperature. The optimum range was 2 to 5°C above saturation temperature. Closer approach to saturated steam temperature resulted in a very large increase in boiler heating demand and a rapid increase in cooler duty beyond cooler capacity. Greater than 5°C superheat on the cooler outlet resulted in reduced boiler power demand and air coolant requirements, as would be expected. However, loop pressure equilibrated at a considerably higher value, indicating lower gas flow rate and higher knallgas concentration - despite the maintenance of a fixed temperature difference between hot and cold legs.

Examination of Metallurgical Samples²

Metallurgical examination of zirconium and titanium alloys, 430 stainless steel samples exposed to the recombiner environment for 2500-2800 hrs indicated that no single material tested was a suitable substitute for Inconel in all parts of the recombiner system. Earlier tests had indicated that stainless steel was subject to stress corrosion.¹

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Fig. 3. Bed Outlet Temperature Vs. Electrolysis Current.

Specimens of iodide zirconium, Zircaloy-2, a Zr-15 Nb alloy, iodide titanium, A-45 titanium, A-110-AT titanium, and type 430 (modified) stainless steel were located in three parts of the loop: boiler liquid, boiler vapor, and downstream from recombiner bed.

Hydrogen pickup and corrosion measurements are tabulated in Appendix D.

Zirconium

Hydrogen analyses disclosed some hydrogen absorption in all the zirconium materials in all sections of the recombiner loop. Hydrogen pickup was appreciable in the recombiner section, but moderate in the boiler sections, except for the Zr-15 Nb alloy specimens which had been exposed also in another recombiner experiment. Thickness change measurements disclosed that only Zircaloy-2 of all the materials tested did not corrode in any of the sections. It was concluded that iodide zirconium and Zircaloy-2 may be usable in the boiler sections of the recombiner loop, containing caustic.

Titanium

Considerable hydrogen pickup occurred in the titanium materials from all sections of the loop. The hydrogen absorption was greatest in the boiler liquid section (250 to 6700 ppm) and least in the recombiner section (50 - 350 ppm). Pretreatment of titanium A-45 in UO_2SO_4 solution appeared to have little or no retarding effect on hydrogen pickup, for specimens exposed in the recombiner section.

All the titanium materials were corrosively attacked in the boiler section, particularly in the boiler liquid (20 - 50 mpy), while corrosion in the recombiner section was nil.

Titanium was concluded to be an unsuitable material of construction for this application.

Modified 430 Stainless Steel

Small amounts of hydrogen were absorbed by the 430 stainless steel in the boiler sections; no significant pickup occurred in the recombiner section (0 to 8 ppm).

Corrosion of 430 stainless steel in the boiler section ranged up to 45 mpy while corrosion in the recombiner section was nil.

Modified 430 stainless steel was rated acceptable as a material of construction for the recombiner section only.

Performance of Inconel as Material of Construction

Precise data on corrosion of Inconel during 2500 hrs operation are lacking. Visual examination of the segmented 2 in. diameter Inconel pipe following the experiment revealed no cracks or obvious defects. All Inconel welds had been stress-relieved at 1150 - 1200°F in a helium atmosphere following fabrication.

CONCLUSIONS AND RECOMMENDATIONS

Operation of the natural circulation recombiner for over 2500 hrs established the concept as a workable, relatively trouble-free method for the catalytic recombination of hydrogen and oxygen at high pressure.

Steady-state knallgas concentration at 0.75 scfm recombination rate was about 8.0 mol %, representing about 20% reaction per pass. The reason for the low fractional reaction was not established, and a better catalyst might be available. Oxygen in 20 psia excess of stoichiometric gas can be used on the basis of this work to maintain an oxidizing atmosphere. Larger excesses appeared to be detrimental; smaller excesses were adequate for relatively short periods to take care of corrosion demand for oxygen. Losses to corrosion became important at low concentrations.

Loop pressure, knallgas concentration, and electric heating and air cooling requirements were most strongly influenced by the choice of cooler outlet temperature. A temperature 2 to 5°C above saturation temperature was optimum as a control point.

Specimens of zirconium and titanium alloys and a modified 430 stainless steel exposed in the system indicated that two zirconium alloys were suitable for boiler fabrication and the 430 stainless steel could be used in the recombiner section. Titanium alloys were unsuitable for either application.

Inconel, from which the loop was fabricated, performed very well. Stress-relief of the assembly was probably an important factor in this application for a high temperature caustic environment.

An extremely slow pressure rise which occurred over a period of several hundred hours operation was not explained. The possibility exists that initial excess oxygen was consumed in corrosion and that the pressure rise was due to a rise in hydrogen pressure. Alternatively catalyst effectiveness may have decreased slightly.

In order to follow properly the dynamics of the natural circulation system, it is recommended that any future experiment provide a means of determining hydrogen concentration - by direct sampling, or other means.

A method for introducing knallgas to a test system, which method did not introduce uncertainties in saturation conditions due to high electrolyte concentration, would be desirable. Most conceivable applications of the natural circulation recombiner would not be burdened by the complication of large (varying) boiling point elevations.

REFERENCES

- (1) P. H. Harley, High Pressure Recombiner Loop Progress Report, ORNL-CF-57-1-90 (1957).
- (2) P. L. Rittenhouse, Examination of Natural Circulation High Pressure Recombiner Loop Specimens, HRP-MET-100 (October 8, 1959).

APPENDIX A

Automatic Safety Devices^(a)

<u>Unsafe Condition</u>	<u>Indication</u>	<u>Sensing Element</u>	<u>Protective Device^(b)</u>
1) Gross leak or rupture	Rapid pressure decrease	Foxboro Dynalog	Manually adjustable pressure switch; usual setting: 1300 psi.
2) Slow leak	a) Drop in boiler level b) Increase in boiler wall temperature	Boiler level instrument Thermocouples under boiler strip heaters	Boiler level instrument relay. Adjustable limit switch-temperature indicating device (Simplytrol).
3) Loss of cooling air or inadequate cooling air supply	Increase in temperature of knallgas-steam effluent of cooler	Thermocouple in cooler effluent	Relay in temperature controller, set for 375°C.
4) High knallgas generation rate	High amperage to electrolytic cells	Indicating ammeter	Adjustable limit switch on ammeter.
5) Boiler heater failure (resulting in decrease in steam diluent concentration)	Low temperature-boiler liquid	Thermocouple	Relay in temperature controller, set for 310°C.
6) Loss of recombination	a) Increase in temperature of superheater wall (due to increase in electrical resistance heat input) b) Increase in total pressure	a) Thermocouple attached to pipe wall b) Foxboro Dynalog b') None required b'') None required	a) Adjustable limit switch-temperature indicating device (Simplytrol). b) Manually adjustable pressure switch; usual setting 1640 psi. b') Rupture disc (1) rated at 2400 psi. b'') Rupture discs (2) rated at 3500 psi.

(a) Any single device can effect complete shutdown of the loop by cutting off electric power to electrolytic cells and all electrical resistance heaters.

(b) The protective device listed for each unsafe condition is the primary device. Usually, however, maloperation results in a combination of unsafe conditions.

APPENDIX BOperation Log

<u>Period</u>	<u>Operations</u>
Sept. 2, 1958	Construction of loop proper completed.
Sept. 3-Sept. 18, 1958	Pressure tested loop for leaks, installed thermal insulation, calibrated instruments.
Sept. 19-Oct. 6, 1958	East electrolytic cell developed a leak at flange. Stopped leak, increased boiler heater capacity. Miscellaneous instrument difficulties.
Oct. 7-Oct. 14, 1958	Operated without excess O ₂ , then with <40 psi excess O ₂ to 1000 amps. Shut down when west cell shorted.
Oct. 15-Oct. 23, 1958	West cell electrically disconnected; east cell only in operation. Increased trim size of air coolant valve. Operation smooth at 2000 amps. Chemical analysis of boiler sample: 4% KOH.
Oct. 24-Nov. 5, 1958	Recharged loop with stratified layers: 18% KOH in cells, tap water in boiler. Operated with 130 psia excess O ₂ . Calculated (Knallgas) was 7.2% at 1400 amps.
Nov. 6-Nov. 24, 1958	Period of intermittent operation; 5°C superheat resulted in stable operation; operation at 13°C and 18°C superheat was unstable. Formation of pin-hole in pressure element shut loop down.
Nov. 25, 1958-Jan. 9, 1959	Procured and installed new pressure element.
Jan. 12-Jan. 13, 1959	Intermittent operation.
Jan. 14-Feb. 1, 1959	Operation at 1500-1600 amps with gradual rise in total pressure which shut loop down after 472 hrs continuous operation.
Feb. 2-Feb. 5, 1959	Chemical analysis of boiler sample: 4% KOH. Operated short time with 50 psia excess oxygen before shut down on high pressure.
Feb. 6-Mar. 14, 1959	Continuous operation, except for two brief scheduled shutdowns.
Mar. 15-Apr. 2, 1959	Intentional shut down.
Apr. 3-May 5, 1959	Continuous operation, for a total of 780 hrs, with 20-25 psia excess oxygen. Boiler (KOH) at end: 3.5%. Total time logged by the loop during recombining operation: 2556 hrs.

APPENDIX CCalculation of Gas Composition by Energy Balance

Basis: Using data from steady-state operation with no electrical superheat, calculate temperature rise across catalyst bed from an assumed total gas flow rate. Calculated rise must agree with observed ΔT (35°C).

From data taken at (cell) time 2534.9 hrs -

$$\begin{aligned}
 P_{\text{total}} &= 61.3\% = 1547 \text{ psia} \\
 -P_{\text{steam}} \text{ at } 308.5^\circ\text{C} &= \frac{1402 \text{ psia}}{\text{(based on } \Delta T_{\text{b.p.}} \text{ for 4\% KOH at } 310.5^\circ\text{C)}} \\
 P_{\text{Kgas}} + P_{\text{XSO}_2} &= 145 \text{ psia} \\
 -P_{\text{XSO}_2} &= -20 \text{ psia (charged at cell time 1776.1 hrs)} \\
 P_{\text{Kgas}} &= 125 \text{ psia}
 \end{aligned}$$

$$[K_{\text{gas}}] = \frac{P_{\text{Kgas}}}{P_{\text{total}}} = \frac{125 \text{ psia}}{1547 \text{ psia}} = 8.1 \text{ mol } \%$$

Gas Composition

$$[H_2] = \frac{(2/3)(125 \text{ psia})}{1547 \text{ psia}} = 5.37 \text{ mol } \%$$

$$[O_2] = \frac{(1/3)(125 \text{ psia})}{1547 \text{ psia}} = 3.95 \text{ mol } \%$$

$$[H_2O] = 100 - 5.37 - 3.95 = 90.68 \text{ mol } \%$$

Measured temperature drop for 3 ft downstream of catalyst bed was zero, therefore, heat loss from the system at the catalyst bed is considered to be negligible.

$$Q_{\text{recomb.}} = N_{\text{gas}} C_{p_{\text{gas}}} \Delta T_{\text{bed}}$$

$$\text{or } \Delta T = \frac{Q_{\text{recomb.}}}{N_{\text{gas}} C_{p_{\text{gas}}}}$$

$$C_{p^*} \text{ for } O_2 \text{ at 4 atm and } 600^\circ\text{K} = 3.865 \times 0.0621 = 0.240 \frac{\text{cal}}{\text{g}^\circ\text{K}} = 7.7 \frac{\text{cal}}{\text{g-mol}^\circ\text{K}}$$

$$C_{p^*} \text{ for } H_2 \text{ at 5.7 atm and } 600^\circ\text{K} = 3.53 \times 0.986 = 3.48 \frac{\text{cal}}{\text{g}^\circ\text{K}} = 7.09 \frac{\text{cal}}{\text{g-mol}^\circ\text{K}}$$

$$C_p \text{ for } H_2O \text{ at 1400 psia and } 600^\circ\text{K} = 1.08 \frac{\text{Btu}}{\text{lb}^\circ\text{F}} = 19.5 \frac{\text{cal}}{\text{g-mol}^\circ\text{K}}$$

$$N_{H_2 \text{ burned}} = (0.98 \text{ generation efficiency})(1810 \text{ amp}) \frac{0.5 \text{ gm-mol } H_2}{26.8 \text{ amp-hr}} \frac{\text{hr}}{60 \text{ min}}$$

$$= 0.551 \frac{\text{g-mols } H_2}{\text{min}}$$

$$Q_{\text{recomb.}} = -58,500 \frac{\text{cal}}{\text{g-mol}} \times \frac{0.551 \text{ g-mol}}{\text{min}} = -32,200 \frac{\text{cal}}{\text{min}}$$

$$\Delta T = \frac{Q_{\text{recomb.}}}{(NCp)_{H_2} + (NCp)_{O_2} + (NCp)_{H_2O}}$$

Assume a total flow of 40 g-mols/min of gas -

$$\Delta T(40) = \frac{32,200 \text{ cal/min}}{[(40)(0.0537) - 0.551] \frac{7.09 \text{ cal}}{\text{g-mol } ^\circ K} + [(40)(0.0395) - 0.275] \frac{7.7 \text{ cal}}{\text{g-mol } ^\circ K} + [(40)(0.9068) + 0.551] \times \frac{19.5 \text{ cal}}{\text{g-mol } ^\circ K}}$$

$$\Delta T_{40} = 43.6^\circ K$$

$$\text{Therefore, } T_{\text{final}} = 315^\circ + 44 = 359^\circ C$$

$$\text{Similarly, } \Delta T_{60} = 29.1^\circ K$$

$$\text{and } T'_{\text{final}} = 344^\circ C$$

$$\text{Also } \Delta T_{50} = 34.9$$

$$\text{and } T''_{\text{final}} = 350^\circ C - \text{in agreement with observed temperature.}$$

Therefore, the flow rate is 50 g-mols gas/minute. Figure C1, Bed Outlet Temperature Vs. Flow Rate, indicates the approximate sensitivity of this trial-and-error solution for flow rate.

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Fig. C1. Bed Outlet Temperature Vs. Flow Rate.

APPENDIX D*

Table D1. Hydrogen Contents of Recombiner Loop Specimens

Specimen Position	Series ¹	Hydrogen Content-ppm							Type 430 Stainless Steel
		Iodide Zr	Zircaloy-2	Zr-15Nb	Iodide Ti	Titanium A-45		Titanium A-110-At	
Control	--	24	60	44	92	27	130 ²	73	1
	B	28	63	46	1800	1100	---	4500	47
Boiler Liquid	C	47	79	72	3020	250	---	500	27
	A	38	110	200	3810	620	---	6740	--
	B	30	78	62	120	51	---	1400	14
Boiler Vapor	C	34	72	98	220	350	---	480	19
	A	63	51	300	240	460	---	1680	6
	B	8000	140	90	140	70	---	190	9
Recombiner	C	1350	200	150	430	190	170 180	360	2
	A	--- ³	230	1630	180	120	---	120	1

*Table taken from memo HRP-MET-100 by P. L. Rittenhouse

¹'B' series - 310-hour high pressure recombiner loop.²

'C' series - 2500-hour natural circulation recombiner loop.¹

'A' series - Received both exposures.

²Pretreated in UO₂SO₄. H₂ pickup not usually observed under conditions of pretreatment.

³Specimen lost in 310-hour loop.

Table D2. Corrosion of Recombiner Loop Specimens

Specimen Position	Series ¹	Thickness Change--inches						
		Iodide Zr	Zircaloy-2	Zr-15Nb	Iodide Ti	Titanium A-45	Titanium A-110-AT	Type 430 Stainless Steel
Boiler Liquid	C	None	None	0.015	0.020	0.012	0.015	0.015
	A	None	None	0.045	0.030	0.020	0.025	0.025
Boiler Vapor	C	None	None	0.020	None	None	0.002	None
	A	None	None	0.100	0.005	0.013	0.010	0.010
Recombiner	C	0.015	None	None	None	None	None	None
	A	3	None	0.010	None	None	None	None

*Table taken from memo HRP-MET-100 by P. L. Rittenhouse

¹'C' series--2500-hour natural circulation recombiter loop.¹

'A' series--310-hour high-pressure recombiter loop² plus 'C' series exposure.

²Estimated corrosion. Only a fragment of a 0.120 in. specimen remained.

³Severe corrosion caused loss of the specimen.

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